PB-217 621

LOW-LEVEL COUNTING BY LIQUID SCINTILLATION II APPLICATIONS OF EMULSIONS IN TRITIUM COUNTING

A.A. Moghissi, et al

Southwestern Radiological Health Laboratory
Las Vegas, Nevada

16 October 1969

DISTRIBUTED BY:

NTIS

National Technical Information Service
U. S. DEPARTMENT OF COMMERCE
5285 Port Royal Road, Springfield Va. 22151

PB 217 621

International Journal of Applied Radiation and Isotopes, 1970, Vol. 21, pp. 319-327. Pergamon Press. Printed in Northern Ireland

Low-Level Counting by Liquid Scintillation—II

APPLICATIONS OF EMULSIONS IN TRITIUM COUNTING

ROBERT LIEBERMAN
Southeastern Radiological Health Laboratory P.O. Box 61, Montgomery, Alabama, U.S.A.

and

A. A. MOGHISSI
Southwestern Radiological Health Laboratory, P.O. Box 15027, Las Vegas, Nevada, U.S.A.

(*Received* 16 *October* 1969)

The evaluation of numerous detergents for application in low-level liquid scintillation counting of tritium in an emulsion system is presented. In a typical operating mode of the selected system having a background of 9 counts/min, one count/min above background corresponds to 200 pCi/l. of water. This increased sensitivity is achieved by the application of a new detergent Triton N101. The described system is stable and reproducible.

LE COMPTAGE DE SCINTILLATION LIQUIDE À BAS NIVEAU—II
LES APPLICATIONS DES EMULSIONS AU COMPTAGE DU TRITIUM

On présente l'évaluation de nombreux détersifs en vu de leur application au comptage de scintillation liquide à bas niveau du tritium dans un système d'émulsion. Dans une façon typique de fonctionnement du système choisi ayant un compte de base de 9 pulsations/min, une pulsation/min au delà du compte de base correspond à 200 pCi/l. d'eau. On achève cette augmentation de sensibilité par l'application d'un nouveau détersif, Triton N101. Le système ici décrit est stable et reproduisible.

FLÜSSIGSZINTILLATIONSZÄHLUNG GERINGER AKTIVITÄTSMENGEN—II
ANWENDUNG VON EMULSIONEN FÜR DIE TRITIUMZÄHLUNG

Zahlreiche oberflächenaktive Produkte sind für die Anwendung in der Flüssigszintillationszählung von geringen Aktivitätsmengen von Tritium untersucht worden. Bei einer typischen Arbeitsweise des ausgewählten Systems mit einer Nullrate von 9 Impulsen/min, entspricht ein Impuls/min. über die Nullrate 200 pCi/l. Wasser. Diese erhöhte Empfpindlichkeit ist durch die Verwendung eines neuen oberflächenaktiven Produktes, Triton N101, erreicht worden. Das beschriebene System ist stabil und reproduzierbar.

СЧЕТ ИЗЛУЧЕНИЯ МАЛОЙ ИНТЕНСИВНОСТИ ЖИДКОСТНОЙ СЦИНТИЛЛЯЦИЕЙ—II
ИСПОЛЬЗОВАНИЕ ЭМУЛЬСИИ В ТРИТИЕВОМ СЧЕТЕ

Дается оценка ряда детергентов для использования в счете излучения тритий малой интенсивности в эмульсионной системе при помощи жидкостной сцинтилляции. В типачном рабочем режиме избранной системы, имеющей фон в 9 счет/мин., один фоновый счет в минуту соответствует 200 ч. Ci/l.ч. воды. Эта повышенная чувствительность достигается использованием нового детергента—Тритона N101. Описываемая система устойчива и воспроизводима.

In a recent paper[1] a method for the low-level counting of tritium using an advanced dioxane-based liquid was reported. Although this system has been operated satisfactorily for several years, the sensitivity of such a solution is limited due to competition between the naphthalene and water with regard to solubility in the dioxane. There is presently no foreseeable concept to considerably increase the sensitivity of this system nor is any other solvent with

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
U S Department of Commerce
Springfield VA 22151

9

superior performance known to replace dioxane for homogeneous counting of water.

In recent years heterogeneous counting systems have gained in popularity. The suspension of water using a silica powder (Cab-O-Sil)[2,3] as well as the suspension of a scintillator in water[4,5] have been described. The production of thixotropic mixtures[6–9] and plastic gels[10] is primarily useful for suspension of solids but could also be used to introduce water in organic solvents.

The suspension of water in aromatic solvents using a detergent is by far the most promising technique for low-level counting of tritium. MEADE and STIGLITZ[11] first introduced Triton X-100, a nonionic detergent, for suspension of aqueous samples. Although other detergents were proposed previously,[8,11,12] Triton X-100 has found a wide acceptance. PATTERSON and GREENE[13] used a toluene:Triton ratio of 2:1 for preparation of a scintillation suspension which could hold up to 23 per cent water. BENSON[14] used a similar solution and pointed out instabilities associated with the detergent-scintillation liquid mixture. In particular, he proposed that samples should be heated to 40° prior to refrigeration in order to achieve reproducible results. VAN DER LAARSE[15] undertook a systematic study of Triton X-100 and proposed an optimum toluene-Triton ratio of 2:1. This mixture could incorporate up to 20 per cent water. WILLIAMS[16] compared dioxane-based solutions and a toluene-Triton-water scintillator (45·5:24·5:30) and concluded that the Triton system was superior to homogeneous counting for low-level counting of tritium. Considering the large number of detergents available, it is surprising that no attempt has been made to evaluate them for application in low-level counting of tritium.

It has been shown recently that p-bis-(o-methylstyryl)-benzene (bis-MSB) is a superior secondary solute[16,17,1] as compared to p-bis-2-(5-phenyloxazolyl) benzene (POPOP) and its dimethyl derivative (DMPOPOP). It has long been established that p-xylene[18] exhibited a higher relative pulse height (RPH) than any other solvent. The melting point of p-xylene (13·2°C), however, made it unsuitable for application in refrigerated systems. The development of new multiplier phototubes and the associated ambient temperature operation of liquid scintillation counters permit the application of p-xylene.

Following is an evaluation of several detergents using bis-MSB and p-xylene as components of the scintillation liquid.

EXPERIMENTAL

Material

A Beckman liquid scintillation counting system was used in this investigation. All scintillators were received from Pilot Chemicals of Watertown, Mass. Toluene and p-xylene (MP 12–14°C) were obtained from Matheson, Coleman and Bell of Cincinnati, Ohio. The scintillation solution (SS) in all cases consisted of 7 g PPO and 1·5 g bis-MSB per liter of toluene and p-xylene, respectively, except where otherwise specified.

Polyethylene vials were received from Packard Instrument Co. of Downers Grove, Ill. (PEP), Beckman instruments of Fullerton, Calif. (PEB) and Nuclear Chicago of Des Plaines, Ill. (PEN). Nylon vials (NC) were also received from Nuclear Chicago.

Tritiated water used in the experiments was calibrated with an NBS tritiated water standard using the method of internal standard with identical system properties.[17] Detergents were received from manufacturers listed in Tables 3 and 4.

Sample preparation for counting was carried out by premixing the detergents and scintillation solvents containing the fluors. The calculated amount of solvent-detergent mixture was added to the vials, followed by addition of water to give the desired volume percent of water. The samples were placed in the scintillation counter and allowed to stand in the changer compartment for 24 hr before counting. The scintillation counter is kept in a light-controlled room with the samples only exposed to a red light during loading and unloading from the instrument.

PROCEDURE AND RESULTS

Counting vials

Counting vial characteristics are an important factor in low-level counting, e.g. it has been established that glass vials are inferior to plastic vials for tritium counting.[1] For dioxane-based scintillation liquids, it has been found[1]

TABLE 1. Loss of compounds through liquid scintillation vials (mg/day)

	Volume	p-xylene	Toluene	Dioxane	H_2O
PEN	25	128	187	4·1	0·5
PEP	25	120	192	3·2	0·1
PEB	20	77	101	1·2	0·3
NC	25	5·3	10	15	34

that PEN vials are the best polyethylene vials presently available. In suspension counting the migration of aromatic solvents and the associated possible change in counting efficiency are the major problems associated with counting vials.

In order that the migration of components of various scintillation mixtures could be established, vials were filled with p-xylene, toluene, and water, respectively. The containers were kept for 30–45 days in a balance room, where temperature was maintained at approximately 22°C, and their weight loss was determined daily during working days. The average weight loss as determined for triplicate samples is shown in Table 1.

PEB vials have a volume of 20 ml and are thus[1] inferior to other larger volume vials tested for low-level counting. Although the migration of organic solvents through the NC vials was slow, water affected these vials considerably. After only a few days NC vials containing water changed their shape and were unuseable in the automatic sample changer. PEP vials had migration properties similar to PEN vials. After a few hours in contact with toluene or xylene, however, they swelled and could not be used in the automatic sample changer. Subsequently, PEN vials were selected and used.

Selection of emulsion system

In a first set of experiments 5 ml of water containing a known amount of tritium were mixed with 20 ml of a SS-detergent mixture. The ratio of SS to detergent was varied. The ratios used were 1, 2, 3 and 4:1, and the resulting emulsion was counted to achieve 0·5 per cent counting error at the ninety-five percent confidence level. The counting efficiency E (cpm/dpm) was then multiplied by the water volume M (ml) and the product was used to compare the results. In Table 2 the maximum $E \cdot M$ value and the ratio of SS to detergent at which $E \cdot M$ reached a maximum are recorded. It can be seen that several detergents have $E \cdot M$ values close to that of Triton X-100. Maximum $E \cdot M$ values were obtained with emulsions which were transparent or slightly hazy to visible light. Low $E \cdot M$ values were generally observed with milky mixtures and

TABLE 2. Performance of detergent: p-xylene scintillation mixtures containing 20 per cent water

Detergent	Maximum $E \times M$(ml)	R*	Detergent type†
Triton N-101	1·41	2	1
Igepal CO-210	0·12	1	1
Igepal CO-430	0·28	1	1
Igepal CO-630	0·98	2	1
Igepal CO-710	1·14	3	1
Igepal CO-850	0·92	2	1
Triton X-100	1·19	4	1
Gafac RM-510	0·76	2	3
Igepal BL-225	0·04	1	4
Tween 60	0·67	1	5
Pluronic L-35	0·92	2	6
Pluronic L-61	0·28	1	6
Pluronic L-62	1·18	2	6
Pluronic L-92	0·62	1	6
Pluronic L-121	0·46	1	6

* Ratio of p-xylene/detergent at maximum $E \cdot M$.

† Classification of detergents used:

1. Alkyl phenyl polyethylene glycol ether
2. Oleic acid-ethylene oxide condensate
3. Ethoxylated alkyl phenyl phosphate
4. Ethoxylated fatty alcohol
5. Polyoxyethylene sorbitan monstearate
6. Polyoxyethylene-polyoxypropylene condensation product
7. Bibliography Ref. (19).
8. Ethoxylated sodium lauryl sulfate
9. Alkyl aryl polyether.

with mixtures which had separated into two phases.

From Table 2 it was established that detergent types 1 and 6 (for classification of detergents see Table 2†) resulted in the highest $E \cdot M$ values.

It was also determined which individual detergents within these two groups resulted in the highest $E \cdot M$ values. Further studies, not described in detail here, showed that only alkyl-phenyl-polyethylene-glycol-ether detergents (type 1) were capable of emulsifying higher water concentrations. These experiments indicated that with this type detergent a SS: detergent ratio of 2·5 to 3:1 could hold 40–50 per cent water.

In a second set of experiments the same procedure as for the first set was repeated, except the water concentration was increased to 40 per cent and the SS:detergent ratio was kept

TABLE 3. Results of detergent screening study using a p-xylene-detergent mixture (2·75:1 volume ratio) containing 40 per cent water

Detergent trade name	$E \times M$(ml)	Manufacturer
Triton N-101	2·39	Rohm and Haas Co.
Triton X-100	1·98	Rohm and Haas Co.
Sterox NJ	2·29	Monsanto Co.
Synthrapol GP	2·15	I.C.I.
Hyonic PE 90	2·20	Nopco Chemical Co.
Renex 698	2·28	Atlas Chemical Co.
Makon M-10	2·08	Stepan Chemical Co.
Hodag E-10	2·10	Hodag Chemical Co.
Solar NPO	2·05	Swift and Company

TABLE 4(a). Detergents with clear appearance between 22–30°C

Detergent trade name	Detergent type*	Manufacturer
Igepal RC-630	1	G.A.F.Corp.
Igepal CO-610	1	G.A.F. Corp.
Igepal CO-710	1	G.A.F. Corp.
Igepal CO-850	1	G.A.F. Corp.
Sterox DJ	1	Monsanto Co.
Makon M-8	1	Stepan Chemical Co.
Pluronic L-62	6	Wyandotte Chemical Co.
Retzanol NP 120	1	Retzloff Chemical Co.
Retzanol NP-150	1	Retzloff Chemical Co.
Retzanol NP-200	1	Retzloff Chemical Co.
Retzanol NP-300	1	Retzloff Chemical Co.
Retzanol NP-500	1	Retzloff Chemical Co.
Poly-Tergent B-300	1	Olin Chemicals
Retzanol NP-100	1	Retzloff Chemical Co.
Hyonic PE-100	1	Nopco Chemical Co.
Igepal CO-630	1	G.A.F. Corp.
Tergitol NPX	1	Union Carbide Co.
Conco NI-100	1	Continental Chemical Co.
Conco NIX-100	1	Continental Chemical Co.
Renex 690	1	Atlas Chemical Co.
Solar NP-100	1	Swift and Company
Synthrapol PWS	1	I.C.I.
Synthrapol KB	1	I.C.I.

* See Table 2†. (p-xylene detergent mixture (2.75:1 volume ratio) containing 40 per cent water.)

TABLE 4(b). Detergents with clear appearance at 10–18°C

Detergent trade name	Detergent type*	Manufacturer
Renex 688	1	Atlas Chemical Co.
Igepal CO-430	1	G.A.F. Corp.

* See Table 2†. (p-xylene detergent mixture (2·75:1 volume ratio) containing 40 per cent water.)

TABLE 4(c). Detergent with opaque appearance or producing two phase between 10–30°C

Detergent trade name	Detergent type*	Manufacturer
Emulphogene BC-720	4	G.A.F. Corp.
Kyro EOB	4	Proctor and Gamble
Sterox-66C	4	Monsanto Co.
Sterox-66L	4	Monsanto Co.
Tween-60	5	Atlas Chemical Co.
Conco Sulfate	8	Continental Chemical Co.
Triton CF-10	9	Rohm and Haas
3 M FC-176	7	3 M Corp.
3 M FC-184	7	3 M Corp.
3 M FC-188	7	3 M Corp.
Pluronic L-35	6	Wyandotte Chemical Co.
Pluronic L-61	6	Wyandotte Chemical Co.
Pluronic L-92	6	Wyandotte Chemical Co.
Pluronic L-121	6	Wyandotte Chemical Co.
Lubrol 90	2	I.C.I.
Igepal CO-210	1	G.A.F. Corp.
Igepal BL-225	4	G.A.F. Corp.
Igepal DM-530	9	G.A.F. Corp.
Igepal DM-730	9	G.A.F. Corp.
Igepal LO-630	9	G.A.F. Corp.
Igepal AL-630	9	G.A.F. Corp.

* See Table 2†. (p-xylene detergent mixture (2·75:1 volume ratio) containing 40 per cent water).

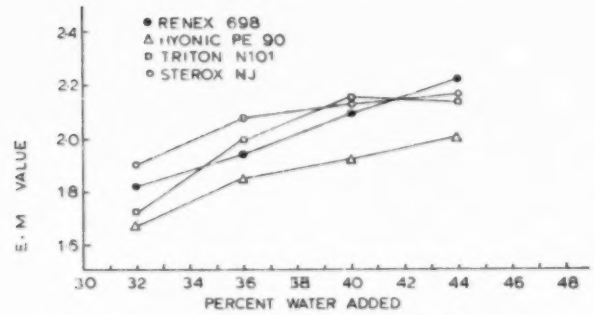

FIG. 1. Performance of four detergents for tritium counting. p-Xylene contained 7 g PPO and 1·5 g bis-MSB/l and was mixed with detergent in volume ratio of 2·75:1.

at 2·75:1. Detergents with clear appearance above and below room temperature as well as the performance of the resulting mixture are shown in Table 3. Tables 4(a) and 4(b) show detergents with transparent appearance above and below room temperature. Table 4(c) lists those detergents that formed opaque or two-phased mixtures resulting in low counting efficiencies at the temperature range of 10–30°C. It is realized that appearance may not necessarily be correlated to the performance of an emulsion but its use in this study as a screening procedure seemed to be justified.

As a result of these investigations four detergents with an $E \cdot M$ value above 2·2 were selected and further studies were conducted to establish the optimum water concentration. In order that a wider water concentration range could be employed the SS to detergent ratio was decreased to 2·5:1.

From Fig. 1 it can be seen that Renex 698, Triton N-101 and Sterox NJ exhibit similar performance. Renex 698 showed higher $E \cdot M$ value at 44 per cent water than the other three. The temperature range at which mixtures with this ratio were stable was, however, too small and in the order of 2–3°C. This temperature range was regarded as a serious limitation for application of this, otherwise efficient, emulsion system. Although all these detergents had similar properties at approximately 40 per cent water concentration, one of them, Triton N-101 was selected for further study. The optimum SS to Triton N-101 ratio for several water concentrations was subsequently investigated. From Fig. 2 it can be seen that a ratio of 2·75:1 results in the highest $E \cdot M$ values for water concentrations above 30 per cent and thus represents the optimum ratio. In practical application, as mentioned, the temperature range at which the emulsion is stable is an important factor. The temperature range of relevant mixtures is shown in Table 5. In order that a convenient operational temperature range could be used, 40 per cent water concentration was selected, although under controlled conditions higher concentrations could be tolerated.

A comparison between Triton N-101 and Triton X-100 using p-xylene and toluene as solvents is shown in Fig. 3. It can be seen that Triton X-100-toluene system is similar in performance to the Triton N-101-p-xylene system up to approximately 30 per cent of water. For higher water concentrations the Triton N-101-p-xylene mixture becomes, however, superior.

As a result of these investigations the following suspension system was selected for routine use: p-xylene containing 7 g PPO and 1·5 g bis-MSB, mixed with Triton N-101 in a volume ratio of 2·75:1. To 15 ml of this solution was added 10 ml of water. The counting efficiency of this system is 22·4 per cent at a background of 9·0 cpm or 24·3 per cent at a background of 15 cpm. This mixture is stable between 17–25°C.

The best Triton X-100 system consisted of toluene containing 7 g PPO and 1·5 g bis-MSB/l and mixed with Triton X-100 in a ratio

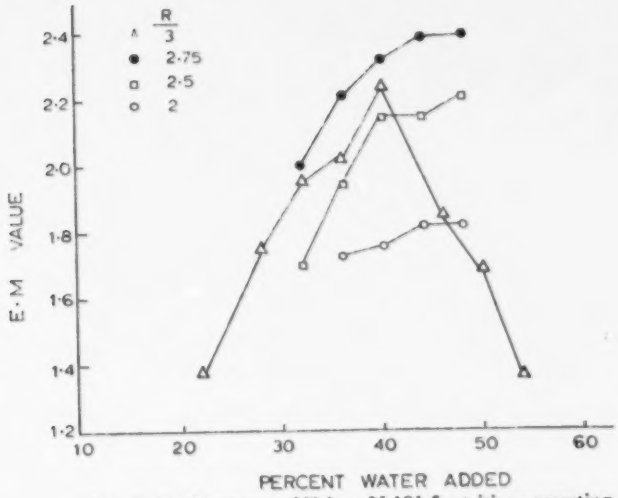

FIG. 2. Performance of Triton N-101 for tritium counting. *R* is the ratio of p-xylene (containing 7 g PPO and 1·5 g bis-MSB/1) to detergent.

of 2·75:1. Seventeen ml of this system could incorporate 8 ml of water resulting in a counting efficiency of 24·0 per cent at a background of 9 cpm or 26·4 per cent at a background of 15 cpm. This mixture is stable between 19–23°C.

STABILITY OF EMULSION SYSTEMS

Several authors have reported changes in counting efficiency associated with emulsion systems[14–16] and proposed methods to increase the reproducibility of these systems. Numerous experiments during the present investigation were carried out to determine changes in counting efficiency with time and the analytical precision attainable with the selected system.

Aside from short-term changes similar to those described for dioxane-based systems, the reproducibility of the method was within the limits of errors associated with pipetting and counting.

For the proposed Triton N-101 xylene mixture, it was found that if water was added first to the scintillation vial, followed by addition of the scintillation solution, the reproducibility varied by as much as 5 per cent. Better reproducibility was achieved when the order of addition was reversed, i.e. adding the scintillation solution to the vial first and followed by the addition of the aqueous sample. The emulsion mixtures described by VAN DER LAARSE[15] and WILLIAMS[16] were repeated adding the scintillation solution first in the vials and reproducible results were obtained from their systems. In search for an explanation for the discrepancy between these and previously

TABLE 5. Useful temperature range of suspension systems

Designation	Ratio of solvent to detergent (S/D)	% Water	Temperature range (°C)
Triton N-101-p-xylene	2·75	40	18–26
Triton N-101-p-xylene	2·75	44	20–26
Triton N-101-p-xylene	2·75	48	22–26
Triton X-100-toluene	2·75	32	19–23

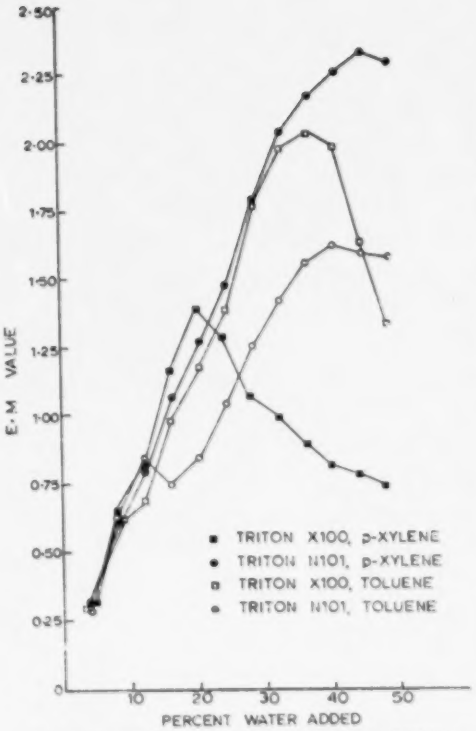

FIG. 3. Performance of Triton N-101 and Triton X-100 mixtures. The solvent contained 7 g PPO and 1·5 g bis-MSB/l and was mixed with the detergent in a volume ratio of 2·75:1.

described results, it was found that whereas our samples were counted at around 20°C, the authors mentioned above used a refrigerated liquid scintillation counter. Apparently the temperature change causes irreproducible results. It is possible that the routine followed in this laboratory of allowing the emulsion samples to stand for 24 hr before counting may also contribute to better reproducibility.

Phosphorescence and chemiluminescence

The accuracy of a background dominant counting system depends upon the stability of background. Phosphorescence and chemiluminescence are two phenomena contributing to background and its instability. The phosphorescence decay in several scintillation mixtures has been studied by LYON *et al.*[18] These investigations indicate that phosphorescence of emulsion systems is severe, although considerably less pronounced than in dioxane-based mixtures.

With elevation of temperatures, the half-life of deexcitation is reduced.[18] The Triton N-101 mixture, e.g. could be totally deexcited by refluxing, while heating the mixture at 40°C had only a limited effect on deexcitation. Also, a small chemiluminescence similar to that described for a dioxane mixture[1] could be observed with the Triton N-101 and Triton X-100 mixtures.

The results of these experiments and the complexity of the nature of phosphorescence and chemiluminescence suggest the avoidance of excitation whenever possible. It has been made a matter of routine procedure in our laboratory to carry out all operations, involving the scintillation liquid, under red light.

Comparison between suspension systems for low-level counting

There seems to be a controversy[18] regarding the performance of various Triton X-100 suspension systems for low-level counting of tritium. The major reason for this controversy is the method of representation of the data. MOGHISSI *et al.*[1] proposed the application of the minimum limit of detection at one-minute counting time and 68 per cent confidence level the *Y*-value for comparison of low-level counting systems.

$$Y = \frac{\sqrt{B}}{2{\cdot}22 \,.\, E \,.\, M}$$

where B is the background in cpm.
E is the efficiency in cpm/dpm, and
M is the volume of water in ml.

In order that the controversy could be solved, the emulsions proposed by WILLIAMS[18] and VAN DER LAARSE[15] were prepared and counted. In addition the same mixtures were prepared and the concentration of PPO was increased and POPOP was replaced by bis-MSB. As a matter of comparison the optimum Triton X-100 and Triton N-101 system as found during this investigation were also included. All experiments were carried out at room temperature. Because of reasons described under "Stability of Emulsion Systems" sample

TABLE 6. Performance of emulsions for low-level counting of tritium

Reference	Solvent (*S*)	PPO (g/l S)	POPOP (g/l S)	bis-MSB (g/l S)	Detergent (*D*)	S/D	Water (ml)	*E*% (*B* = 9·0)	*E*% (*B* = 15·0)	*Y* (pCi/ml)
WILLIAMS[16]	Toluene	7	0·5	—	Triton X-100	1·85	7·5	22·0	24·0	0·82
WILLIAMS (revised)	Toluene	7	—	1·5	Triton X-100	1·85	7·5	22·6	24·5	0·80
VAN DER LAARSE[16]	Toluene	4	0·3	—	Triton X-100	2·20	5·0	22·0	24·7	1·23
VAN DER LAARSE[16] (revised)	Toluene	7	—	1·5	Triton X-100	2·20	5·0	25·1	28·6	1·07
This paper	Toluene	7	—	1·5	Triton X-100	2·75	8·0	24·0	26·4	0·70
This paper	P-xylene	7	—	1·5	Triton N-101	2·75	10·0	22·4	24·1	0·60

were not heated as proposed by Williams. All experiments involving a scintillation mixture were carried out under red light. All samples were kept for 24 hr in a dark room prior to counting. Counting efficiencies were recorded for a background of 15·0 cpm which corresponds to the highest efficiency obtainable. Efficiencies were also recorded for a background of 9·0 cpm which represents the efficiency of the optimum *Y*-value. The backgrounds were obtained using duplicate samples for 100 min counting time. Results of these experiments are shown in Table 6.

DISCUSSION

From the present investigation it can be seen that a number of detergents may be advantageously used for low-level counting of tritium. A *Y*-value of 0·6 pCi/ml is obtainable with the proposed Triton N-101 system under routine laboratory conditions. If temperature is rigidly controlled, even lower values may be obtained. The proposed system is reproducible if water is added as the last component of the mixture. This method does not require heat or other treatments. During the present investigations a more efficient Triton X-100 system, with the *Y*-value of 0·7 pCi/ml was found. Low and reproducible background counts were obtained by avoiding light excitation of the scintillation mixture.

Apparently the suspension characteristics of Triton X-100 are more compatible with toluene than p-xylene as indicated by lower *Y*-values of Triton X-100-toluene system.

As detergents are to some extent quenchers, their concentration should be kept to a minimum. The lower *Y*-value of Triton X-100 system described in this paper as compared to that proposed by Williams is primarily due to the reduced detergent concentration. It is conceivable that other detergents may be found which incorporate higher water concentrations and thus reduce the *Y*-value.

The increased sensitivity of detergent systems is associated with the necessity of application of these mixtures at a limited temperature range. Outside of this range, a reduction in counting efficiency or a phase separation may occur. The proposed system has the advantage of ambient temperature operation. Numerous samples have been counted in our laboratory in an instrument with no provision for temperature control. If the room temperature deviates more than the recommended temperature range of the mixture, a temperature controlled instrument should be used.

Acknowledgements—The authors wish to express their appreciation for the technical assistance of Mae W. Williams in performing the laboratory experiments.

Mention of commercial products used in connection with work reported in this article does not constitute an endorsement by the Public Health Service.

REFERENCES

1. Moghissi A. A., Kelley H. L., Regnier J. E. and Carter M. W. *Int. J. appl. Radiat. Isotopes* **20,** 145 (1969).
2. Ott D. G., Richmond C. R., Trujillo T. T. and Foreman H. *Nucleonics* **17,** 106 (1959).
3. Gordon C. F. and Wolfe A. L. *Analyt. Chem.* **32,** 574 (1960).
4. Steinberg D. *Nature, Lond.* **182,** 740 (1958).
5. Steinberg D. *ibid.* **183,** 1254 (1959).
6. Funt F. N. *Nucleonics* **14,** 83 (1956).
7. White C. G. and Helf S. *ibid.* **14,** 47 (1956).
8. Shapira J. and Perkins W. H. *Science* **131,** 414 (1960).
9. Helf S., White C. G. and Shelley R. N. *Analyt. Chem.* **32,** 239 (1960).
10. Bollinger J. N., Marlow W. A., Register J. W. and Johnson D. E. *Analyt. Chem.* **18,** 193 (1967).
11. Meade R. C. and Stiglitz R. A. *Int. J. appl. Radiat. Isotopes* **13,** 11 (1962).
12. Kinnory D. S., Kanabrocki D. S., Greco J., Veatch R. L., Kaplan E. and Oester Y. T. *Liquid Scintillation Counting* p. 223. Paragon Press, Oxford (1959).
13. Patterson M. S. and Greene R. C. *Analyt. Chem.* **37,** 854 (1965).
14. Benson R. C. *Analyt. Chem.* **38,** 1353 (1966).
15. Van der Laarse J. D. *Int. J. appl. Radiat. Isotopes* **18,** 485 (1967).
16. Williams P. H. *ibid.* **19,** 377 (1968).
17. Moghissi A. A. and Carter M. W. *Analyt. Chem.* **40,** 812 (1968).
18. Lyon R. J. and Moghissi A. A. (to be published).
19. Guenthner R. A. and Vietor M. L. Surface active materials from perfluorocarboxylic and perfluorosulfonic acids. Presented at the *Am. chem. Soc. Mtg* (1961).

AU GAFB ALA (711625) K 100